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PATENT

Trimethylolalkanes and process for preparing them

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The object of the present invention is the preparation
of trimethylolalkanes and, in particular, of tri-
10 methylolpropane.

It has long been known that pentaerythritol can be
prepared by condensing formaldehyde with acetaldehyde
in a basic medium.

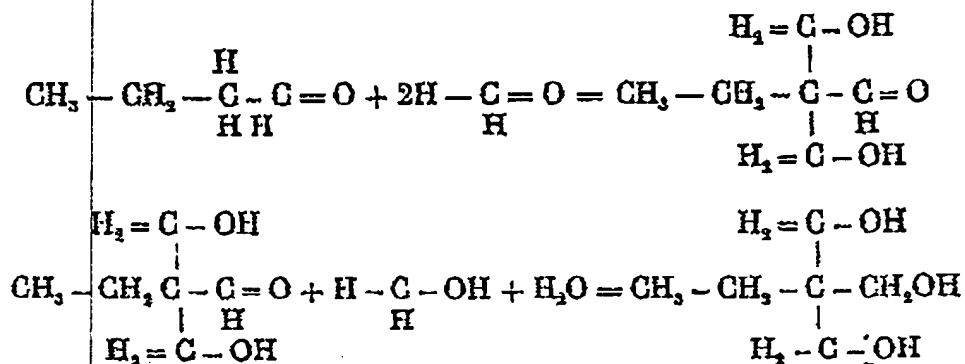
15 The present invention consists, for preparing the
substances indicated above, in performing similar
reactions but starting from aldehydes other than
acetaldehyde, so as to prepare compounds containing
20 three methylol groups attached to the same carbon, the
fourth valency of the carbon being satisfied by a
radical originating from a saturated hydrocarbon.

In particular, using normal-butyric aldehyde as base
25 aldehyde, rather than acetaldehyde, the product, in
accordance with the invention, is trimethylolpropane.

In this case, the two hydrogen atoms borne by the
carbon adjacent to the functional group each react with
30 one molecule of formaldehyde to give two methylol
radicals, while the aldehyde group undergoes
combination with a third molecule of formaldehyde to
form another methylol group, in accordance with the
conventional mechanism of a Cannizzaro reaction. At the
35 same time formic acid is released, and is immediately
neutralized by the base in whose presence the reaction
is conducted.

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Overall, the conversions may be written schematically as follows:



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In practice it will be possible to operate by reacting one molecule of normal-butyric aldehyde with three molecules of formaldehyde in the presence of lime at a temperature in the region of 60°C and under atmospheric pressure.

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The yields are in the region of 66%. To make, for example, 100 kg of trimethylolpropane in the course of a certain operation involves consumption of the following:

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101.7 kg of formaldehyde and
81.3 kg of butyric aldehyde.

Following purification, the trimethylolpropane is in the form of a relatively hard solid which has the appearance of a greyish white technical-grade paraffin.

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Its melting point is 53°C and it distils at 194°C under 2 mm pressure. This substance very rapidly absorbs atmospheric water vapour and undergoes superficial liquefaction on contact with the air.

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By vacuum distillation it is relatively easy to separate trimethylolpropane from its dimer, which is analogous to dipentaerythritol; the operation is more convenient than for pentaerythritol since, in the course of this distillation, or more precisely this

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concentration, the higher condensation products, analogous to sugars, are completely destroyed by carbonization.

5 As a consequence it is possible with ease to vary the industrial quality of trimethylolpropanes, since varying the quantity of heat supplied for the vacuum concentration automatically involves purification to a greater or lesser extent.

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The purity of trimethylolpropane can be assessed with a fair degree of exactitude in the course of a nitration test. With a pure product, the nitrate has a melting point of 51.6°, a density of 1.48, and the yield reaches 91.5%.

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A less pure product gives, in addition, a certain amount of nitrate derivative of the dimer, in the form of an oily liquid, having the appearance of nitro-glycerine.

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It will of course be possible to make numerous modifications without departing from the scope of the invention; in particular, it will be possible to replace the butyric aldehyde by other homologues of acetaldehyde.

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CLAIMS

1. Process for preparing trimethylolalkanes, and especially trimethylolpropane, which consists in
5 reacting formaldehyde with a higher homologue of acetaldehyde and, in particular, normal-butyric aldehyde.
2. Specific embodiments of this process which are
10 applicable to the preparation of different technical grades of trimethylol, in which a greater or lesser degree of purity is determined by the manner in which the final concentration is conducted, the said final
15 concentration being accompanied by destruction of the sugars to an extent which increases in proportion with the duration of the carbonization.

la qualité industrielle des triméthylol-propanes, car, en agissant sur la quantité de chaleur fournie pour la concentration sous vide, on réalise automatiquement une purification plus ou moins poussée.

Le degré de pureté d'un triméthylol-propane peut être apprécié assez exactement au cours d'un essai de nitration. Avec un produit pur, le nitrate a un point de fusion de 51,6 °, une densité de 1,48 et le rendement atteint 91,5 %.

Un produit moins pur donne, en plus, une certaine quantité de dérivé nitré du dimère, sous la forme d'un liquide huileux, ayant l'aspect de la nitro-glycérine.

On pourra apporter, bien entendu, de nombreuses modifications sans sortir du domaine de l'invention, en particulier on pourra remplacer l'aldéhyde butyrique par d'autres homologues de l'acétaldéhyde.

RÉSUMÉ

1° Procédé de préparation de triméthylol-alcanes, et notamment du triméthylol-propane, consistant à faire réagir du formol avec un homologue supérieur de l'acétaldéhyde et notamment de l'aldéhyde butyrique normal;

2° Modes de réalisation particuliers de ce procédé applicables à la préparation de différentes qualités de triméthylol techniques, dans lesquels un plus ou moins grand degré de pureté est déterminé par la façon dont est conduite la concentration finale, laquelle s'accompagne d'une destruction des sucres, d'autant plus complète que la durée de la carbonisation est plus longue.

ÉTAT FRANÇAIS REPRÉSENTÉ PAR M. LE MINISTRE DE LA
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